

## $\mu$ -Hexathiometadiphosphato-bis[(1,4,7,-10,13,16-hexaoxacyclooctadecane- $\kappa^6$ O)-rubidium] acetonitrile disolvate

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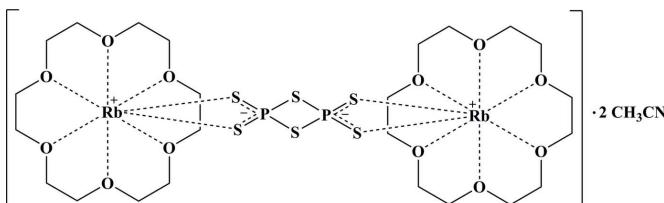
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Key indicators: single-crystal X-ray study;  $T = 223\text{ K}$ ; mean  $\sigma(\text{C}-\text{C}) = 0.007\text{ \AA}$ ;  $R$  factor = 0.047;  $wR$  factor = 0.074; data-to-parameter ratio = 12.8.

The asymmetric unit of the title compound,  $[\text{Rb}_2(\text{P}_2\text{S}_6)\cdot(\text{C}_{12}\text{H}_{24}\text{O}_6)_2]\cdot 2\text{CH}_3\text{CN}$ , contains one half of an  $[\text{Rb}(18\text{-crown-6})_2][\text{P}_2\text{S}_6]$  unit and one acetonitrile solvent molecule. The  $[\text{Rb}(18\text{-crown-6})_2][\text{P}_2\text{S}_6]$  unit is completed by inversion symmetry. Its  $\text{Rb}^+$  ion is situated near the centre of the macrocyclic cavity, but is displaced by  $0.8972(1)\text{ \AA}$  from the O atoms of the crown in the direction of the  $[\text{P}_2\text{S}_6]^{2-}$  moiety. The overall coordination number of the cation is eight, defined by the six crown ether O atoms and by two terminal S atoms of the  $[\text{P}_2\text{S}_6]^{2-}$  anion. The hexathiometadiphosphate anion is built up from two tetrahedral  $\text{PS}_4$  units joined together by a common edge. The crystal structure is characterized by alternating layers of  $[\text{Rb}(18\text{-crown-6})_2][\text{P}_2\text{S}_6]$  and acetonitrile solvent molecules stacked along [010].

### Related literature

For the synthesis of hexathiometadiphosphates, see: Thilo & Ladwig (1962). For the crystal structures of hexathiometadiphosphates, see: Toffoli *et al.* (1978); Brockner *et al.* (1985). For the crystal structures of alkali crown ether hexathiometadiphosphates, see: Gjikaj *et al.* (2005, 2006).



### Experimental

#### Crystal data

$[\text{Rb}_2(\text{P}_2\text{S}_6)(\text{C}_{12}\text{H}_{24}\text{O}_6)_2]\cdot 2\text{CH}_3\text{CN}$	$V = 2323.5(4)\text{ \AA}^3$
$M_r = 1035.98$	$Z = 2$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 8.2261(9)\text{ \AA}$	$\mu = 2.50\text{ mm}^{-1}$
$b = 17.1054(15)\text{ \AA}$	$T = 223\text{ K}$
$c = 16.5895(18)\text{ \AA}$	$0.29 \times 0.26 \times 0.22\text{ mm}$
$\beta = 95.520(9)^\circ$	

#### Data collection

Stoe IPDSII diffractometer	26056 measured reflections
Absorption correction: numerical ( <i>X-SHAPE</i> and <i>X-RED</i> ; Stoe & Cie, 1999, 2001)	4400 independent reflections
$T_{\min} = 0.490$ , $T_{\max} = 0.577$	3450 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.092$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.047$	343 parameters
$wR(F^2) = 0.074$	All H-atom parameters refined
$S = 1.15$	$\Delta\rho_{\max} = 0.50\text{ e \AA}^{-3}$
4400 reflections	$\Delta\rho_{\min} = -0.28\text{ e \AA}^{-3}$

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *SHELXL97*, *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2785).

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# supplementary materials

*Acta Cryst.* (2013). E69, m689 [doi:10.1107/S1600536813032121]

## **$\mu$ -Hexathiometadiphosphato-bis[(1,4,7,10,13,16-hexaoxacyclooctadecane- $\kappa^6$ O)rubidium] acetonitrile disolvate**

**Mimoza Gjikaj, Niels-Patrick Pook and Flora Qarri**

### **1. Comment**

The first thiophosphates with empirical formula  $MPS_3$  ( $M = \text{Na, Ag and Tl}$ ), were described by Thilo & Ladwig (1962). However, the crystal structure determination of  $\text{Ag}_2\text{P}_2\text{S}_6$  proved the presence of isolated  $\text{P}_2\text{S}_6^{2-}$  anions (Toffoli *et al.*, 1978). The first alkali metal hexathiometadiphosphate structures,  $M_2\text{P}_2\text{S}_6$  ( $M = \text{K and Cs}$ ), were determined by Brockner *et al.* (1985). By using crown ether as complexing agent, alkali hexathiometadiphosphates,  $M_2\text{P}_2\text{S}_6$  ( $M = \text{Na and K}$ ), can be dissolved in  $\text{CH}_3\text{CN}$ . Such crown-ether-stabilized thiophosphates were obtained in crystalline form by Gjikaj *et al.* (2005, 2006).

The structure of the title compound,  $[\text{Rb}(18\text{-crown-6})_2[\text{P}_2\text{S}_6]\text{2CH}_3\text{CN}]$ , is isotopic with  $[\text{K}(18\text{-crown-6})_2[\text{P}_2\text{S}_6]\text{2CH}_3\text{CN}$  (Gjikaj *et al.*, 2005) and is characterized by alternating layers of  $[\text{Rb}(18\text{-crown-6})_2[\text{P}_2\text{S}_6]$  and acetonitrile molecules stacked along [010]. The asymmetric unit consists of one half of an  $[\text{Rb}(18\text{-crown-6})_2[\text{P}_2\text{S}_6]]$  unit, which is located on a centre of inversion, and is completed by one acetonitrile molecule. The eightfold coordination environment of rubidium is defined by the six crown ether oxygen atoms and by two terminal sulfur atoms of the hexathiometadiphosphate anion (Fig. 2). The hexathiometadiphosphate anion is built up by two edge-sharing  $\text{PS}_4$  units, each with a tetrahedral arrangement. The P–S bond lengths are ranging from 1.9630 (13) to 2.1419 (13) Å. All bond lengths and angles are comparable to those found for  $[\text{K}(18\text{-crown-6})_2[\text{P}_2\text{S}_6]\text{2CH}_3\text{CN}$  (Gjikaj *et al.*, 2005).

### **2. Experimental**

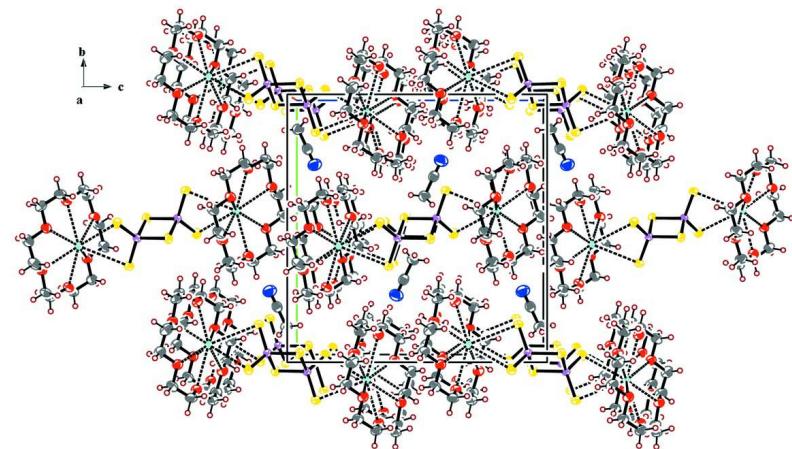
Rubidium hexathiometadiphosphate was prepared by high-temperature element synthesis using the procedure reported by Brockner *et al.* (1985). A solution of bis[(1,4,7,10,13,16-hexaoxacyclooctadecane- $\kappa^6$ O)rubidium] hexathiometadiphosphate was obtained by adding rubidium hexathiometadiphosphate to a solution of 18-crown-6 in dry acetonitrile.

#### **2.1. Refinement**

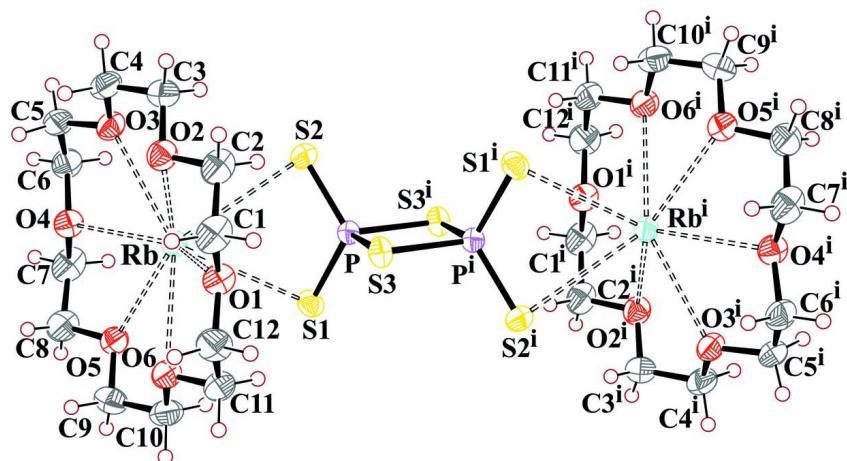
All hydrogen atoms were located in a difference Fourier map and were refined isotropically with no restraints.

#### **Computing details**

Data collection: *X-Area* (Stoe & Cie, 2002); cell refinement: *X-Area* (Stoe & Cie, 2002); data reduction: *X-Area* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

**Figure 1**

The crystal structure of the title compound. Displacement ellipsoids at the 50% probability level. The Rb—O and Rb—S bonds are shown as dashed lines.

**Figure 2**

The molecular structure of the title compound with atom labels and 50% probability displacement ellipsoids. The Rb—O and Rb—S bonds are shown as dashed lines. [Symmetry codes: (i)  $-x, -y + 1, -z - 1$ .]

**$\mu$ -Hexathiometadiphosphato-bis[(1,4,7,10,13,16-hexaoxacyclooctadecane- $\kappa^6$ O)rubidium] acetonitrile disolvate***Crystal data*

[Rb <sub>2</sub> (P <sub>2</sub> S <sub>6</sub> )(C <sub>12</sub> H <sub>24</sub> O <sub>6</sub> ) <sub>2</sub> ]·2C <sub>2</sub> H <sub>3</sub> N	$F(000) = 1064$
$M_r = 1035.98$	block, yellow
Monoclinic, $P2_1/c$	$D_x = 1.481 \text{ Mg m}^{-3}$
Hall symbol: -P 2yn	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$a = 8.2261 (9) \text{ \AA}$	Cell parameters from 41621 reflections
$b = 17.1054 (15) \text{ \AA}$	$\theta = 2.4\text{--}25.7^\circ$
$c = 16.5895 (18) \text{ \AA}$	$\mu = 2.50 \text{ mm}^{-1}$
$\beta = 95.520 (9)^\circ$	$T = 223 \text{ K}$
$V = 2323.5 (4) \text{ \AA}^3$	Block, yellow
$Z = 2$	$0.29 \times 0.26 \times 0.22 \text{ mm}$

*Data collection*

Stoe IPDSII	26056 measured reflections
diffractometer	4400 independent reflections
Radiation source: fine-focus sealed tube	3450 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{\text{int}} = 0.092$
$\omega$ -scans	$\theta_{\text{max}} = 25.7^\circ, \theta_{\text{min}} = 2.4^\circ$
Absorption correction: numerical ( <i>X-SHAPE</i> and <i>X-RED</i> ; Stoe & Cie, 1999, 2001)	$h = -10 \rightarrow 9$
$T_{\text{min}} = 0.490, T_{\text{max}} = 0.577$	$k = -20 \rightarrow 20$
	$l = -20 \rightarrow 20$

*Refinement*

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.047$	All H-atom parameters refined
$wR(F^2) = 0.074$	$w = 1/[\sigma^2(F_o^2) + (0.0188P)^2 + 1.1961P]$
$S = 1.15$	where $P = (F_o^2 + 2F_c^2)/3$
4400 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
343 parameters	$\Delta\rho_{\text{max}} = 0.50 \text{ e \AA}^{-3}$
0 restraints	$\Delta\rho_{\text{min}} = -0.28 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

*Special details*

**Geometry.** All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement.** Refinement of  $F^2$  against ALL reflections. The weighted  $R$ -factor  $wR$  and goodness of fit  $S$  are based on  $F^2$ , conventional  $R$ -factors  $R$  are based on  $F$ , with  $F$  set to zero for negative  $F^2$ . The threshold expression of  $F^2 > \sigma(F^2)$  is used only for calculating  $R$ -factors(gt) etc. and is not relevant to the choice of reflections for refinement.  $R$ -factors based on  $F^2$  are statistically about twice as large as those based on  $F$ , and  $R$ -factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Rb	0.01688 (5)	0.43425 (2)	-0.19589 (2)	0.03401 (10)
S1	0.01382 (16)	0.35946 (6)	-0.38945 (6)	0.0451 (3)
S2	-0.20728 (13)	0.51995 (6)	-0.36048 (6)	0.0374 (2)
S3	0.14602 (13)	0.53670 (6)	-0.44812 (5)	0.0348 (2)

P	-0.05739 (12)	0.46416 (5)	-0.42619 (5)	0.0290 (2)
O1	0.2674 (4)	0.54136 (15)	-0.23640 (16)	0.0410 (6)
O2	0.0200 (4)	0.60293 (16)	-0.14890 (16)	0.0415 (7)
O3	-0.2169 (4)	0.50147 (15)	-0.09188 (15)	0.0406 (6)
O4	-0.1161 (3)	0.34506 (15)	-0.06497 (16)	0.0394 (6)
O5	0.1325 (4)	0.27789 (15)	-0.14684 (15)	0.0412 (7)
O6	0.3663 (4)	0.38439 (16)	-0.19993 (16)	0.0416 (7)
C1	0.2628 (7)	0.6212 (3)	-0.2123 (3)	0.0509 (12)
C2	0.0893 (7)	0.6461 (3)	-0.2104 (3)	0.0482 (11)
C3	-0.1428 (7)	0.6269 (3)	-0.1389 (3)	0.0499 (11)
C4	-0.2044 (7)	0.5824 (3)	-0.0707 (3)	0.0479 (11)
C5	-0.2661 (7)	0.4551 (3)	-0.0274 (3)	0.0525 (12)
C6	-0.2744 (6)	0.3710 (3)	-0.0527 (3)	0.0540 (12)
C7	-0.1060 (6)	0.2626 (2)	-0.0796 (3)	0.0473 (11)
C8	0.0674 (6)	0.2404 (2)	-0.0800 (3)	0.0435 (10)
C9	0.3003 (6)	0.2593 (3)	-0.1507 (3)	0.0457 (10)
C10	0.3625 (7)	0.3033 (3)	-0.2188 (3)	0.0476 (11)
C11	0.4266 (6)	0.4296 (3)	-0.2630 (3)	0.0499 (11)
C12	0.4300 (6)	0.5138 (3)	-0.2389 (3)	0.0513 (11)
N13	-0.5159 (6)	0.2422 (3)	-0.4116 (3)	0.0750 (13)
C14	-0.5164 (6)	0.3022 (3)	-0.4407 (3)	0.0478 (10)
C15	-0.5201 (8)	0.3788 (3)	-0.4778 (4)	0.0580 (13)
H1A	0.324 (6)	0.633 (3)	-0.160 (3)	0.054 (13)*
H1B	0.323 (7)	0.652 (3)	-0.252 (3)	0.073 (16)*
H2A	0.026 (5)	0.639 (2)	-0.261 (3)	0.044 (12)*
H2B	0.098 (6)	0.702 (3)	-0.197 (3)	0.062 (14)*
H3A	-0.146 (6)	0.683 (3)	-0.125 (3)	0.065 (14)*
H3B	-0.212 (6)	0.618 (2)	-0.192 (3)	0.050 (13)*
H4A	-0.303 (6)	0.603 (3)	-0.056 (3)	0.054 (13)*
H4B	-0.129 (6)	0.590 (3)	-0.018 (3)	0.056 (13)*
H5A	-0.373 (7)	0.473 (3)	-0.016 (3)	0.077 (17)*
H5B	-0.179 (6)	0.463 (2)	0.019 (3)	0.052 (13)*
H6A	-0.308 (5)	0.342 (2)	-0.008 (3)	0.047 (12)*
H6B	-0.341 (6)	0.367 (3)	-0.106 (3)	0.062 (15)*
H7A	-0.143 (5)	0.237 (2)	-0.036 (2)	0.041 (11)*
H7B	-0.179 (7)	0.253 (3)	-0.132 (3)	0.077 (17)*
H8A	0.078 (5)	0.189 (3)	-0.083 (2)	0.041 (11)*
H8B	0.137 (5)	0.259 (2)	-0.027 (3)	0.050 (12)*
H9A	0.319 (6)	0.204 (3)	-0.158 (3)	0.056 (13)*
H9B	0.364 (5)	0.271 (2)	-0.097 (3)	0.046 (12)*
H10A	0.300 (6)	0.296 (3)	-0.267 (3)	0.050 (13)*
H10B	0.470 (6)	0.287 (3)	-0.229 (3)	0.054 (13)*
H11A	0.537 (6)	0.413 (3)	-0.269 (3)	0.054 (13)*
H11B	0.353 (6)	0.420 (3)	-0.314 (3)	0.061 (14)*
H12A	0.485 (6)	0.520 (3)	-0.186 (3)	0.052 (13)*
H12B	0.480 (6)	0.543 (2)	-0.282 (3)	0.051 (13)*
H15A	-0.451 (8)	0.411 (4)	-0.446 (4)	0.09 (2)*
H15B	-0.493 (7)	0.374 (3)	-0.531 (4)	0.083 (18)*
H15C	-0.630 (9)	0.396 (4)	-0.485 (4)	0.11 (3)*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Rb	0.0386 (2)	0.03336 (18)	0.03045 (17)	-0.00168 (18)	0.00526 (13)	0.00247 (16)
S1	0.0684 (8)	0.0288 (5)	0.0373 (5)	0.0062 (5)	0.0010 (5)	0.0021 (4)
S2	0.0354 (6)	0.0432 (5)	0.0349 (5)	0.0033 (4)	0.0099 (4)	-0.0027 (4)
S3	0.0333 (5)	0.0429 (5)	0.0282 (4)	-0.0058 (4)	0.0022 (4)	0.0005 (4)
P	0.0323 (6)	0.0286 (4)	0.0263 (4)	0.0002 (4)	0.0043 (4)	0.0010 (4)
O1	0.0347 (16)	0.0398 (15)	0.0487 (15)	-0.0043 (12)	0.0055 (12)	0.0030 (12)
O2	0.0426 (18)	0.0380 (15)	0.0440 (15)	0.0020 (13)	0.0046 (13)	0.0031 (12)
O3	0.0415 (17)	0.0390 (15)	0.0412 (15)	0.0022 (13)	0.0036 (12)	0.0018 (12)
O4	0.0361 (17)	0.0375 (15)	0.0453 (15)	-0.0053 (12)	0.0072 (12)	0.0027 (12)
O5	0.0460 (18)	0.0349 (14)	0.0427 (15)	0.0005 (13)	0.0048 (13)	0.0079 (11)
O6	0.0401 (17)	0.0418 (15)	0.0433 (15)	-0.0003 (13)	0.0063 (13)	0.0048 (12)
C1	0.058 (3)	0.038 (2)	0.058 (3)	-0.017 (2)	0.013 (2)	-0.002 (2)
C2	0.063 (3)	0.034 (2)	0.048 (3)	-0.003 (2)	0.008 (2)	0.0046 (19)
C3	0.056 (3)	0.034 (2)	0.059 (3)	0.005 (2)	0.003 (2)	-0.001 (2)
C4	0.045 (3)	0.047 (3)	0.053 (3)	0.005 (2)	0.008 (2)	-0.0113 (19)
C5	0.045 (3)	0.059 (3)	0.057 (3)	0.011 (2)	0.023 (2)	0.011 (2)
C6	0.036 (3)	0.060 (3)	0.068 (3)	-0.004 (2)	0.016 (2)	0.019 (3)
C7	0.059 (3)	0.033 (2)	0.050 (3)	-0.010 (2)	0.007 (2)	0.0054 (19)
C8	0.058 (3)	0.027 (2)	0.046 (2)	-0.002 (2)	0.009 (2)	0.0046 (17)
C9	0.047 (3)	0.040 (2)	0.050 (3)	0.011 (2)	0.002 (2)	0.0071 (19)
C10	0.052 (3)	0.042 (2)	0.050 (3)	0.010 (2)	0.009 (2)	-0.0009 (19)
C11	0.037 (3)	0.057 (3)	0.057 (3)	0.004 (2)	0.014 (2)	0.015 (2)
C12	0.035 (3)	0.056 (3)	0.064 (3)	-0.008 (2)	0.009 (2)	0.014 (2)
N13	0.086 (4)	0.058 (3)	0.081 (3)	0.012 (3)	0.011 (3)	0.009 (2)
C14	0.043 (3)	0.054 (3)	0.046 (2)	0.006 (2)	0.003 (2)	-0.008 (2)
C15	0.065 (4)	0.050 (3)	0.058 (3)	-0.010 (3)	-0.002 (3)	0.003 (2)

Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

Rb—O1	2.885 (3)	C3—C4	1.491 (7)
Rb—O5	2.928 (3)	C3—H3A	0.98 (5)
Rb—O3	2.939 (3)	C3—H3B	1.01 (5)
Rb—O4	2.949 (3)	C4—H4A	0.93 (5)
Rb—O2	2.988 (3)	C4—H4B	1.02 (5)
Rb—O6	3.005 (3)	C5—C6	1.499 (7)
Rb—S1	3.4544 (11)	C5—H5A	0.97 (6)
Rb—S2	3.4692 (11)	C5—H5B	1.01 (5)
S1—P	1.9630 (13)	C6—H6A	0.96 (4)
S2—P	1.9692 (13)	C6—H6B	1.00 (5)
S3—P <sup>i</sup>	2.1418 (13)	C7—C8	1.477 (7)
S3—P	2.1421 (14)	C7—H7A	0.92 (4)
P—S3 <sup>i</sup>	2.1419 (13)	C7—H7B	1.02 (5)
O1—C12	1.423 (6)	C8—H8A	0.89 (4)
O1—C1	1.424 (5)	C8—H8B	1.05 (4)
O2—C2	1.422 (5)	C9—C10	1.489 (6)
O2—C3	1.425 (6)	C9—H9A	0.97 (5)
O3—C5	1.421 (5)	C9—H9B	1.01 (4)

O3—C4	1.430 (5)	C10—H10A	0.91 (5)
O4—C6	1.409 (5)	C10—H10B	0.96 (5)
O4—C7	1.435 (5)	C11—C12	1.495 (7)
O5—C9	1.423 (5)	C11—H11A	0.97 (5)
O5—C8	1.428 (5)	C11—H11B	1.01 (5)
O6—C10	1.422 (5)	C12—H12A	0.95 (5)
O6—C11	1.428 (5)	C12—H12B	1.00 (4)
C1—C2	1.493 (7)	N13—C14	1.135 (6)
C1—H1A	0.98 (5)	C14—C15	1.446 (7)
C1—H1B	1.01 (5)	C15—H15A	0.92 (7)
C2—H2A	0.96 (4)	C15—H15B	0.93 (6)
C2—H2B	0.97 (5)	C15—H15C	0.95 (7)
O1—Rb—O5	115.13 (8)	C4—C3—H3A	108 (3)
O1—Rb—O3	114.25 (8)	Rb—C3—H3A	160 (3)
O5—Rb—O3	114.06 (7)	O2—C3—H3B	108 (3)
O1—Rb—O4	145.48 (8)	C4—C3—H3B	113 (3)
O5—Rb—O4	57.07 (8)	Rb—C3—H3B	80 (2)
O3—Rb—O4	57.31 (7)	H3A—C3—H3B	108 (4)
O1—Rb—O2	57.26 (8)	O3—C4—C3	109.2 (4)
O5—Rb—O2	144.60 (8)	O3—C4—H4A	112 (3)
O3—Rb—O2	57.22 (8)	C3—C4—H4A	112 (3)
O4—Rb—O2	107.56 (7)	O3—C4—H4B	111 (3)
O1—Rb—O6	57.92 (8)	C3—C4—H4B	111 (3)
O5—Rb—O6	57.38 (8)	H4A—C4—H4B	102 (4)
O3—Rb—O6	143.65 (8)	O3—C5—C6	109.5 (4)
O4—Rb—O6	107.03 (7)	O3—C5—H5A	108 (3)
O2—Rb—O6	107.20 (8)	C6—C5—H5A	110 (3)
O1—Rb—S1	87.72 (6)	O3—C5—H5B	105 (3)
O5—Rb—S1	83.87 (5)	C6—C5—H5B	111 (3)
O3—Rb—S1	138.08 (6)	H5A—C5—H5B	113 (4)
O4—Rb—S1	121.53 (6)	O4—C6—C5	109.0 (4)
O2—Rb—S1	126.82 (5)	O4—C6—H6A	107 (3)
O6—Rb—S1	78.21 (6)	C5—C6—H6A	107 (3)
O1—Rb—S2	83.44 (6)	Rb—C6—H6A	155 (3)
O5—Rb—S2	137.62 (6)	O4—C6—H6B	107 (3)
O3—Rb—S2	88.20 (6)	C5—C6—H6B	109 (3)
O4—Rb—S2	126.26 (6)	Rb—C6—H6B	78 (3)
O2—Rb—S2	77.75 (6)	H6A—C6—H6B	118 (4)
O6—Rb—S2	122.65 (5)	O4—C7—C8	108.9 (4)
S1—Rb—S2	57.93 (3)	O4—C7—H7A	108 (3)
P—S1—Rb	85.75 (4)	C8—C7—H7A	106 (3)
P—S2—Rb	85.25 (4)	Rb—C7—H7A	156 (3)
P <sup>i</sup> —S3—P	87.88 (5)	O4—C7—H7B	105 (3)
S1—P—S2	117.02 (6)	C8—C7—H7B	116 (3)
S1—P—S3 <sup>i</sup>	111.07 (6)	Rb—C7—H7B	82 (3)
S2—P—S3 <sup>i</sup>	111.69 (6)	H7A—C7—H7B	112 (4)
S1—P—S3	111.50 (7)	O5—C8—C7	108.9 (4)
S2—P—S3	110.75 (6)	O5—C8—H8A	111 (3)

S3 <sup>i</sup> —P—S3	92.12 (5)	C7—C8—H8A	111 (3)
S1—P—Rb	63.64 (4)	O5—C8—H8B	108 (2)
S2—P—Rb	64.06 (4)	C7—C8—H8B	111 (2)
S3 <sup>i</sup> —P—Rb	166.70 (5)	H8A—C8—H8B	107 (3)
S3—P—Rb	101.17 (4)	O5—C9—C10	109.1 (4)
C12—O1—C1	112.1 (4)	O5—C9—H9A	113 (3)
C12—O1—Rb	119.4 (2)	C10—C9—H9A	110 (3)
C1—O1—Rb	120.3 (3)	O5—C9—H9B	110 (2)
C2—O2—C3	112.3 (3)	C10—C9—H9B	112 (2)
C2—O2—Rb	108.0 (2)	H9A—C9—H9B	103 (4)
C3—O2—Rb	108.9 (2)	O6—C10—C9	109.1 (4)
C5—O3—C4	112.0 (3)	O6—C10—H10A	109 (3)
C5—O3—Rb	118.3 (2)	C9—C10—H10A	113 (3)
C4—O3—Rb	119.2 (2)	Rb—C10—H10A	78 (3)
C6—O4—C7	113.8 (3)	O6—C10—H10B	109 (3)
C6—O4—Rb	110.9 (2)	C9—C10—H10B	112 (3)
C7—O4—Rb	110.6 (2)	Rb—C10—H10B	160 (3)
C9—O5—C8	111.9 (3)	H10A—C10—H10B	105 (4)
C9—O5—Rb	118.8 (2)	O6—C11—C12	108.9 (4)
C8—O5—Rb	119.3 (2)	O6—C11—H11A	108 (3)
C10—O6—C11	111.6 (3)	C12—C11—H11A	109 (3)
C10—O6—Rb	106.3 (3)	Rb—C11—H11A	160 (3)
C11—O6—Rb	105.3 (2)	O6—C11—H11B	108 (3)
O1—C1—C2	109.4 (4)	C12—C11—H11B	112 (3)
O1—C1—H1A	115 (3)	Rb—C11—H11B	76 (3)
C2—C1—H1A	109 (3)	H11A—C11—H11B	111 (4)
O1—C1—H1B	107 (3)	O1—C12—C11	109.5 (4)
C2—C1—H1B	113 (3)	O1—C12—H12A	108 (3)
H1A—C1—H1B	103 (4)	C11—C12—H12A	110 (3)
O2—C2—C1	108.5 (4)	O1—C12—H12B	108 (3)
O2—C2—H2A	110 (3)	C11—C12—H12B	107 (2)
C1—C2—H2A	113 (3)	H12A—C12—H12B	115 (4)
Rb—C2—H2A	82 (2)	N13—C14—C15	179.0 (6)
O2—C2—H2B	112 (3)	C14—C15—H15A	108 (4)
C1—C2—H2B	103 (3)	C14—C15—H15B	109 (4)
Rb—C2—H2B	162 (3)	H15A—C15—H15B	114 (5)
H2A—C2—H2B	110 (4)	C14—C15—H15C	109 (4)
O2—C3—C4	109.5 (4)	H15A—C15—H15C	115 (6)
O2—C3—H3A	111 (3)	H15B—C15—H15C	102 (5)

Symmetry code: (i)  $-x, -y+1, -z-1$ .